FORMS PTO-1390 (REV 10-96) U S DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE

TRANSMITTAL LETTER TO THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US) CONCERNING A FILING UNDER 35 U.S.C. 371

ATTORNEY'S DOCKET NUMBER	U.S. APPLICATION NO. (If known, see 37 CFR 1.5)
DeLiso 11-3-5-8	60/114,379 09/869441

INTERNATIONAL APPLICATION NO.	INTERNATIONAL FILING DATE	PRIORITY DATE CLAIMED
PCT/US99/29225	09 December 1999	30 December 1998

TITLE OF INVENTION

TANTALA DOPED WAVEGUIDE AND METHOD OF MANUFACTURE

APPLICANT(S) FOR DO/EO/US

Corning Incorporated

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. ☒ This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371.
2. ☐ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C.371.
3. ☐ This express request to begin national examination procedures (35 U.S.C. 371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and PCT Articles 22 and 39(1).
4. ☐ A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date.
5. ☒ A copy of the International Application as filed (35 U.S.C. 371(c)(2))
   a. ☐ is transmitted herewith (required only if not transmitted by the International Bureau).
   b. ☐ has been transmitted by the International Bureau.
   c. ☒ is not required, as the application was filed in the United States Receiving Office (RO/US).
6. ☐ A translation of the International Application into English (35 U.S.C. 371(c)(2)).
7. ☒ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3)).
   a. ☒ are transmitted herewith (required only if not transmitted by the International Bureau).
   b. ☐ have been transmitted by the International Bureau.
   c. ☐ have not been made; however, the time limit for making such amendments has NOT expired.
   d. ☐ have not been made and will not be made.
8. ☐ A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).
9. ☒ An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)).
10. ☐ A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).

Items 11. To 16. Below concern document(s) or information included:

11. ☒ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
12. ☒ An Assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
13. ☐ A FIRST preliminary amendment.
14. ☐ A SECOND or SUBSEQUENT preliminary amendment.
15. ☐ A change of power of attorney and/or address letter.
16. ☐ Other items or information:

U.S. APPLICATION NO. (If known, see 37 CFR 1.5)	INTERNATIONAL APPLICATION NO.	ATTORNEY'S DOCKET NUMBER
60/114,269 09/869441	PCT/US99/29225	DeLiso 11-3-5-8

17. ☒ The following fees are submitted:

BASIC NATIONAL FEE (37 CFR 1.492 (a)(1)-(5):

Search Report has been prepared by the EPO or JPO.....**$840.00**
International preliminary examination fee paid to USPTO (37 CFR 1.482).....**$670.00**
No international preliminary examination fee paid to USPTO (37 CFR 1.482) but international search fee paid to USPTO (37 CFR 1.445(a)(2)).....**$760.00**
Neither international preliminary examination fee (37 CFR 1.482) not international search fee (37 CFR 1.445(a)(2)) paid to USPTO.....**$970.00**
International preliminary examination fee paid to USPTO (37 CFR 1.482) and all claims satisfied provisions of PCT Article 33(2)-(4).....**$96.00**

CALCULATIONS	PTO USE ONLY

ENTER APPROPRIATE BASIC FEE AMOUNT = $ 840.00

Surcharge of **$130.00** for furnishing the oath or declaration later than ☐ 20 ☐ 30 months from the earliest claimed priority date (37 CFR 1.492(e)). $

CLAIMS	NUMBER FILED	NUMBER EXTRA	RATE	
Total claims	22 - 20 =	2	X $18.00	$ 36.00
Independent claims	3 - 3 =		X $80.00	$
MULTIPLE DEPENDANT CLAIM(S) (if applicable)			+ $260.00	$

TOTAL OF ABOVE CALCULATIONS = $ 876.00

Reduction of ½ for filing by small entity, if applicable. Verified Small Entity Statement must also be filed (Note 37 CFR 1.9, 1.27, 1.28) + $

SUBTOTAL = $ 876.00

Processing fee of **$130.00** for furnishing the English translation later than ☐ 20 ☐ 30 months from the earliest claimed priority date (37 CFR 1.492(f)). + $

TOTAL NATIONAL FEE = $ 876.00

Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). **$40.00** per property + $ 40.00

TOTAL FEES ENCLOSED = $ 916.00

Amount to be refunded: $

Charged: $ 916.00

a. ☐ A check in the amount of $ _____ to cover the above fees is enclosed.
b. ☒ Corning Incorporated hereby authorizes use of Deposit Account No. 03-3325 in the amount of $916.00 to cover the above fees. A duplicate copy of this sheet is enclosed.
c. ☒ The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. 03-3325. A duplicate copy of this sheet is enclosed.

NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.

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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

PCT APPLICATION

Applicant: Corning Incorporated

: RESPONSE TO WRITTEN OPINION

Serial No.: PCT/US99/29225

Filing Date: 09 December 1999

For: TANTALA DOPED WAVEGUIDE AND METHOD OF MANUFACTURE

Assistant Commissioner of Patents and Trademarks
Box PCT
Washington, D.C. 20231

Dear Sirs:

In response to the Written Opinion dated 21 September 2000, applicant, in accord with PCT Rule 66.3, asks that the following amendment be entered and considered before issue of the Preliminary Examination Report.

AMENDMENT TO CLAIMS

17. (amended) A glass for use in the core of an optical waveguide fiber comprising: SiO_2; and,

by weight on an oxide basis after consolidation, between about 2% non-crystallized Ta_2O_3 to 5% non-crystallized Ta_2O_3, wherein the attenuation of said optical waveguide fiber at 1550 nm is less than 0.25 dB/km.

Please cancel claim 23.

REMARKS

Amended claim 17 now contains the limitation that attenuation at 1550 nm is less than 0.25 dB/km. Such an attenuation is impossible using the glass disclosed in XP-002141764 because that glass contains 2000 ppm of OH^-. It is well known in the art that such high OH^- concentration is not compatible with low attenuation optical waveguide fiber. See, for example, Electro-optics Handbook, McGraw-Hill, Inc., 1994, Chapter Twelve, Page 12.35, Table 12.2, Figure 12.19. (Copy enclosed)

CONCLUSION

Based on the above Amendments and Remarks, applicant hereby respectfully requests the entry of the above amendments prior to the issuance of the International Preliminary Examination Report.

Replacement pages 15 and 16 of the claims are attached.

Respectfully submitted,

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5 wt% Ta_2O_5 after consolidation, and wherein light attenuation in said optical fiber is less than about 1.8 dB/km at 1550 nm.

15. The optical fiber as claimed in claim 14 wherein said glass core further includes SiO_2 and wherein said optical fiber is substantially free of crystals.

16. The optical fiber as claimed in claim 15 wherein light attenuation in said optical fiber comprises about 0.25 dB/km at 1550 nm.

17. A glass for use in the core of an optical waveguide comprising:

SiO_2; and

by weight on an oxide basis after consolidation, between about 2% non-crystallized Ta_2O_3 to 5% non-crystallized Ta_2O_3, wherein the attenuation of said optical waveguide fiber at 1550 nm is less than 0.25 dB/km.

18. The glass as claimed in claim 17 wherein said core glass is consolidated in a helium atmosphere at a temperature of between about 1600° C to about 2000° C.

19. The glass as claimed in claim 18 wherein said core glass is consolidated in a helium atmosphere at a temperature of between about 1600° C to about 1800° C.

20. The glass as claimed in claim 19 wherein said core glass is consolidated in a helium atmosphere at a temperature of between about 1600° C to about 1650° C.

21. The core glass as claimed in claim 17 wherein said core glass is consolidated in a vacuum atmosphere at a temperature greater than about 1450° C.

22. The core glass as claimed in claim 17 wherein said core glass is bounded by a cladding comprising SiO_2 to form an optical fiber, and wherein light attenuation in said optical fiber is less than about 1.8 dB/km at 1550 nm.

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09/869441

JC03 Rec'd PCT/PTC 26 JUN 2001

TANTALA DOPED WAVEGUIDE AND METHOD OF MANUFACTURE

FIELD OF THE INVENTION

The present invention relates generally to optical waveguide glass having a high index of refraction and a method for manufacturing such optical waveguide glass, and more particularly to a method of doping optical waveguide glass with Ta_2O_5 to produce essentially crystalline free optical waveguide fiber.

While the invention is capable of being carried out using a number of soot collection and doping techniques, it is particularly well suited for use in conjunction with the outside vapor deposition (OVD) process, and will be particularly described in that regard.

BACKGROUND OF THE INVENTION

In the rapidly expanding field of telecommunications, there is an ever-increasing demand for systems that transfer greater amounts of data in shorter periods of time. Accordingly, in the opto-electronics field, there is a continuing need for new optical waveguide systems, and consequently new optical waveguides and new optical waveguide components for meeting the demands of those systems.

Generally speaking, optical waveguide fibers include a core surrounded by a cladding material having a refractive index lower than that of the core. Such optical waveguide fibers are generally composed of silica that is selectively doped with a dopant such a germanium. Although germanium is the principal and most widely used dopant, other dopants such as phosphorous, fluorine, boron and erbium, to name a few, are often used. Germania, however, is most commonly used due to its low melting point and high refractive index in relation to silica.

All dopants, including germania have shortcomings that limit their usefulness to certain applications. Accordingly, as technology improves and the requirements for new applications increases, the requirement for new optical waveguide fiber capable of meeting the demands of these applications increases as well. Such needs provide the incentive to consider the application of new dopants and new methods of doping optical waveguide fibers to meet these demands. In addition, competition is continually driving researchers to develop optical waveguide fibers at lower cost. Because germania costs approximately $1,000 per kilogram, a less expensive dopant capable of providing a higher index of refraction than germania with less of that alternative dopant would be ideal.

One such dopant known to have a high refractive index is tantala. In fact, Ta_2O_5 thin films are widely used in thin-film waveguide lenses and anti-reflective coatings for silicon wafer solar cells. Because of the attractiveness of Ta_2O_5, thin films for integrated optical devices, many researchers have been active in this area. Thin films for integrated optical devices containing Ta_2O_5 are typically fabricated using sputtering techniques and result in measurable losses of about 0.4 dB/cm. In the field of thin-films it is believed that a contributing factor to such high losses is the subsequent heat treatment of thin-films following sputtering. It was found that the heat treatment caused the film to change from amorphous to crystalline. Such a defect, if formed in an optical waveguide fiber, would adversely affect that optical waveguide fiber operating properties and would render the fiber non-functional in an optical waveguide fiber system.

Planar devices have also been fabricated using Ta_2O_5. Ta_2O_5-SiO_2 core glass for such devices is laid down using an electron beam vapor deposition technique. However, the lowest loss observed for such devices has been approximately 0.15 dB/cm or 15,000 dB/km. For optical waveguide fiber, losses of less than approximately 1 dB/km is the target. Thus, neither the thin-films nor the planar optical devices suggest the usefulness of tantala doped silica for optical waveguide fibers.

In view of the foregoing, there is a need for a dopant that, in limited quantities, is capable of providing a high core index of refraction to an optical waveguide fiber. In addition, there exists a need for a dopant that has good non-linear properties, does not adversely impact the mechanical properties of the optical waveguide fiber in which is resides, and exhibits beneficial amplification characteristics. Moreover, there is a need for a method of providing the dopant to an optical waveguide fiber with minimal deviation from present optical fiber manufacturing techniques, thus making it economically feasible and desirable. The low cost of tantala compared to germania, as well as tantala's high index of refraction makes it a promising candidate for such a dopant.

SUMMARY OF THE INVENTION

One aspect of the present invention relates to a method of manufacturing a low loss optical waveguide having a high refractive index core by forming a soot blank which includes Ta_2O_5 and SiO_2, consolidating the soot blank to form a cane under conditions suitable to prevent crystallization of the Ta_2O_5- SiO_2 containing glass and drawing the cane into an optical fiber.

In another aspect, the invention relates to an optical fiber that is manufactured by preparing a soot blank which includes at least Ta_2O_5 and SiO_2, consolidating the soot blank to form a cane under conditions suitable to prevent crystallization of the Ta_2O_5- SiO_2 containing glass and drawing the cane into an optical fiber.

A further aspect of the invention relates to an optical fiber having a high purity glass cladding, and a high refractive index glass core bounded by the cladding. The glass core includes between about 2 to 5 wt% Ta_2O_5, so that light attenuation in the optical fiber is less than about 1.8 dB/km at 1550 nm.

Yet another aspect of the invention relates to a glass for use in the core of the optical waveguide that includes SiO_2 and, by weight on an oxide basis, between about 2% non-crystallized Ta_2O_5, to 5% non-crystallized Ta_2O_5 after consolidation.

The glass and method of the present invention results in a number of advantages over other glasses and methods known in the art. One of the most attractive features of using tantala in the glass for the present invention is its high index of refraction, which is reported to be 2.2 at 632.8 nm. Accordingly, in the glass of the present invention, the same refractive index change can be achieved with a much lower addition of Ta_2O_5 than can be achieved with GeO_2. Moreover, because tantala is far less expensive than germania, there is a significant cost savings resulting from the selection of tantala as a dopant.

Another advantage is the high viscosity of Ta_2O_5-SiO_2 glass, which is a function of the high melting point of tantala. Ta_2O_5 has a melting point of 1887°C while SiO_2 and GeO_2 have melting points of 1715°C and 1116°C, respectively. Accordingly, the high viscosity of tantala silicate glass makes the glass of the present invention a likely candidate for viscosity matching.

Additional advantages of the present invention are that tantalum oxide is chemically stable and insoluble in water, the thermal expansion of glass containing tantala is lower than that of glass containing germania, and the method of the present invention essentially eliminates crystallization within the Ta_2O_5- SiO_2 containing glass during the manufacture of optical waveguides. The latter advantage results in improved optical characteristics.

Additional features and advantages of the invention will be set forth in the detailed description which follows, and in part will be readily apparent to those skilled in the art from the description or recognized by practicing the invention as described in the written description and claims hereof, as well as the appended drawings.

It is to be understood that both the foregoing general description and the following detailed description are merely exemplary of the invention and are intended to provide an overview or framework to understanding the nature and character of the invention as it is claimed.

The accompanying drawings are included to provide a further understanding of the invention and are incorporated in and constitute a part of this specification. The drawings illustrate one or more embodiments of the invention, and together with the description serve to explain the principles and operation of the invention.

BRIEF DESCRIPTION OF THE DRAWINGS

Fig. 1 is a perspective view of an optical fiber manufactured in accordance with the present invention.

Fig. 2 is a cross-section view of the optical fiber of Fig. 1 taken through line 2-2 in Fig. 1.

Fig. 3 is a cross-section view of a Cl_2 reactor of the present invention.

Fig. 4 is a schematic view of a vapor delivery system shown forming a soot blank in accordance with the present invention.

Fig. 5 is a schematic view of a first preferred embodiment of a consolidation furnace of the present invention taken in cross-section.

Fig. 6 is a schematic view of a second preferred embodiment of a consolidation furnace of the present invention taken in cross-section.

Fig. 7 is a photomicrograph of a Ta_2O_5 doped core glass consolidated at 1450°C in a helium atmosphere.

Fig. 8 is a photomicrograph of a Ta_2O_5 doped core glass consolidated at 1450°C in a helium atmosphere.

Fig. 9 is a photomicrograph showing the core-clad interface of Ta_2O_5 doped glass consolidated at 1450°C in a helium atmosphere.

Fig. 10 is a photomicrograph of a Ta_2O_5 doped core glass consolidated at 1550°C in a helium atmosphere.

Fig. 11 is a photomicrograph of a Ta_2O_5 doped core glass consolidated at 1550°C in a helium atmosphere.

Fig. 12 is a photomicrograph of a Ta_2O_5 doped core glass consolidated at 1550°C in a helium atmosphere.

Fig. 13 is a photomicrograph of a Ta_2O_5 doped core glass consolidated at 1450°C in a vacuum atmosphere.

Fig. 14 is a photomicrograph of a Ta_2O_5 doped core glass consolidated at 1550°C in a vacuum atmosphere.

Fig. 15 is a photomicrograph of a Ta_2O_5 doped core glass consolidated at 1650°C in a vacuum atmosphere.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS

The present invention expressly contemplates the manufacture of single-mode optical waveguide fibers, multimode optical waveguide fibers, and planar waveguides regardless of any specific description, drawings, or examples set out herein. In addition, it is anticipated that the present invention can be practiced in conjunction with any of the known optical waveguide processing techniques, including, but not limited to, the outside vapor deposition (OVD) technique, the modified chemical vapor deposition (MCVD) technique, the vertical axial deposition (VAD) technique, the plasma chemical vapor deposition (PCVD) technique, and sol-gel techniques, to name a few. However, for the purposes of this specification, the tantala silicate soot and blanks described herein and shown in the accompanying drawing figures are described as being manufactured using the OVD technique.

Reference will now be made in detail to the present preferred embodiments of the invention, examples of which are illustrated in the accompanying drawings. Wherever possible, the same reference characters will be used throughout the drawings to refer to the same or like parts. An exemplary embodiment of the optical waveguide of the present invention is shown in Fig. 1, and is designated generally throughout by reference character 20.

In accordance with the invention, the present invention for an optical waveguide fiber 20 includes a high purity glass cladding 22 and a high refractive index glass core 24 bonded by the cladding 22. As embodied herein, and depicted in Figs. 1 and 2, high purity glass cladding 22 is predominantly silica, and core 24 includes silica doped with tantala in the desired proportions. Optical waveguide fiber 20 having between about 2 to 5 wt% non-crystalline Ta_2O_5 after consolidation has been demonstrated to exhibit a loss of less than about 1.8 dB/km at 1550 nm. In a preferred embodiment, light attenuation in optical waveguide fiber 20 is less than 0.25 dB/km at 1550 nm.

A preferred embodiment of the method of manufacturing a low-loss optical waveguide having a high refractive index core includes the steps of forming a soot blank which includes Ta_2O_5 and SiO_2, consolidating the soot blank to form a cane under conditions suitable to prevent crystallization of the Ta_2O_5, and drawing the cane into an optical fiber. The Ta_2O_5 can be delivered using chemical vapor deposition techniques known in the art or via liquid delivery. The SiO_2 can similarly be delivered using known chemical vapor deposition techniques or liquid delivery.

An exemplary embodiment of a reactor for use with the chemical vapor deposition technique is shown in Fig. 3. Reactor 26 includes a diffuser 28, a preheat zone 30, and a reaction zone 32. In operation, fragments of tantalum 34 are packed within the preheat zone 30 of reactor 26 and chlorine (Cl_2) gas is flowed through diffuser 28 and over the fragments of tantalum 34 within reactor 26. Reactor 26 includes two separate heater coils (not shown) for the for the preheat zone 30 and reaction zone 32. When the heat in the reaction zone is 350°C or greater, a sufficient quantity of $TaCl_5$ gas is formed in reactor 26 to provide a desired amount of Ta_2O_5 in the soot.

As shown schematically in Fig. 4, $TaCl_5$ gas is delivered from vapor delivery system 36 to a burner assembly 38. The $TaCl_5$ is converted to Ta_2O_5 in the burner flame 40 according to the following reaction:

$$4\ TaCl_5\ (g) + 5\ O_2\ (g) = 2\ Ta_2O_5 + 10\ Cl_2\ (g)$$

Finely divided amorphous Ta_2O_5 containing soot 42 is thereafter projected from the flame for capture and further processing. In a preferred embodiment, soot 42 is captured on a rotating mandrel 46 to form a soot blank 44. The amount of Ta_2O_5 captured on soot blank 44 is determined by the number of lateral passes made by burner assembly 38 along the length of soot blank 44, as well as the flow rate of Cl_2 through reactor 26.

The consolidation furnaces used for consolidating germania silicate blanks manufactured using OVD techniques typically provide temperatures of between 1000°C and 1450°C. Through experimentation, it has been found that such furnaces do not provide the heat necessary to perform the consolidation step without crystallization in the Ta_2O_5-SiO_2 containing glass as required for the present invention. Accordingly, improved consolidation furnaces capable of achieving temperatures in excess of 1450°C are needed for the present invention. The preferred embodiments of such consolidation furnaces are shown schematically in Figs. 5 and 6.

Fig. 5 depicts a first preferred embodiment of the consolidation step of the method of manufacturing a low loss optical waveguide having a high refractory index core. Soot blank 44 is held within consolidation furnace 48 where it is exposed to a gas 50. Gases such as, but not limited to, chlorine, helium, and oxygen, or combinations thereof, are delivered into consolidation furnace 48 to form the atmosphere 52 therein. Presently, the preferred gas, helium, is flowed across soot blank 44 while temperatures within consolidation furnace 48 are preferably elevated to 1600°C or greater. These conditions are maintained within consolidation furnace 48 until the Ta_2O_5-SiO_2 core glass temperatures are maintained at 1600°C or higher for a suitable time to sinter and vitrify the glass. After taking the additional processing steps commonly known to those skilled in the art in optical fiber manufacture, the resulting cane is drawn into an optical fiber. It is anticipated that an optical fiber manufactured from a SiO_2 soot blank containing 2 to 5 wt% Ta_2O_5, and heat treated to a temperature of 1600°C or higher in a flowing helium atmosphere will have an attenuation of less than about 0.25 dB/km at 1550 nm. In a preferred embodiment, the temperature range is approximately 1600°C to 1700°C.

Fig. 6 depicts a second preferred embodiment of consolidation furnace 48 shown supporting soot blank 44. In this embodiment of the present invention, soot blank 44 is heated within a vacuum atmosphere. As used herein, the phrase "vacuum atmosphere" means an atmosphere less than atmospheric pressure. As depicted in Fig. 6, a pump 56 or other pressure-reducing device, removes the air from within consolidation furnace 48, thereby decreasing the pressure therein. As a result, soot blank 44 can be heat treated at temperatures lower than 1600°C to sinter and vitrify soot blank 44. Typically, soot blank 44 is heated to a temperature between 1500°C and 1600°C in a vacuum atmosphere so that the Ta_2O_5-SiO_2 core glass temperatures reach between 1500°C and 1600°C for a sufficient time to result in clear glass which is substantially free of crystals. In a preferred embodiment, the vacuum atmosphere 54 within consolidation furnace 48, exhibits a pressure of less than about 10^{-4} torr. Following the additional processing steps commonly known to those skilled in the art of optical fiber manufacture, the resulting cane is drawn into an optical fiber. An optical fiber manufactured from a soot blank 44 containing SiO_2 and about 2 to 5 wt% Ta_2O_5, and heat treated at temperatures ranging between 1500°C and 1600°C in a vacuum atmosphere having a pressure of less then 10^{-4} torr is expected to exhibit attenuation of less than about 0.25 dB/km at 1550 nm.

A significant advantage of the method of the present invention is the crystalline free consolidation of Ta_2O_5 containing soot blanks. The following examples illustrate the effectiveness of the method of the present invention.

Example 1

A core blank was made by depositing 100 passes of Ta_2O_5-SiO_2 at an analyzed chemical wt% of 5.55 Ta_2O_5, followed by 177 passes of SiO_2. The resulting soot preform specimen was cut into cross-sectional slices approximately 25 millimeters long and approximately 50 to 60 millimeters in diameter. Samples were then fired at a temperature of 1450°C in flowing helium as shown in Figs. 7-9. The scanning electron micrographs (SEMs) of

the core material (Figs. 7 and 8) and the core material below the core-clad interface (Fig. 9) show that crystallization is prevalent in the Ta_2O_5-SiO_2 containing glass. As shown clearly in the fiber section 60 of FIG. 9, the silica cladding 62 is easily distinguished from the Ta_2O_5-SiO_2 containing core 64 as the cladding 62 has consolidated to a clear, amorphous glass. A core-clad interface region 66 is clearly visible between the cladding 62 and core 64.

Example 2

Additional slices of the soot preform specimen described above with respect to Example 1 were heated to 1550°C under a flowing helium atmosphere. The results of this experiment are shown in Figs. 10 and 11. The SEM's again show that the Ta_2O_5 containing core glass depicted in Figs. 10 and 11 contained numerous crystals. In fact, crystallization is so prevalent that increasing the temperature by approximately 100°C does not appear to reduce crystallization as compared to Example 1.

Example 3

An additional slice from the soot preform specimen described in Example 1 above was heat treated in a flowing helium atmosphere to a temperature of 1650°C. As shown in the SEM of Fig. 12, the core sample consolidated to a clear glass having no apparent crystallization.

Example 4

Additional slices of the soot preform specimen described in Example 1 were also fired at temperatures of 1450°C, 1550°C and 1650°C in a vacuum atmosphere of 1×10^{-4} torr. As seen in Fig. 13, the SEM shows that crystallization is present in the Ta_2O_5 containing core glass after heat treatment

at 1450°C. However, at treatment temperatures of 1550°C and 1650°C, as shown in the SEM's of Figs. 14 and 15, respectively, no crystallization occurs in the Ta_2O_5-SiO_2 core glass.

To permit other testing, single-mode step index optical fibers were drawn from other core blanks prepared in a manner substantially similar to that described above with respect to examples 1 – 4. The %.Δ, and attenuation for fibers containing different amounts of Ta_2O_5 by weight percent are shown below in Table 1.

Table I

Results for Single Mode Fibers with Tantala Silicate Core

Sample #	Wt% Ta2O5	Delta (%)	Attenuation @ 1310 nm	Attenuation @ 1380 nm	Attenuation @ 1550 nm
1	2.0	0.25	15.6	29.5	4.3
2	2.0	0.25	33.3	40.6	12.4
3	2.0	0.25	26.7	38.8	11.3
4	2.9	0.31	3.6	16.4	2.25
5	2.9	0.30	2.89	7.26	1.73
6	3.1	0.34	4.3	21.5	2.21
7	4.5	0.50	212.7	175.2	82.4

The consolidation furnace used to heat treat the fibers listed in Table I were standard furnaces commonly used to consolidate GeO_2-SiO_2 optical fiber preforms. Accordingly, the maximum temperature available for consolidation was 1450°C. Thus, the maximum temperature of 1450°C was used to consolidate each of the core blanks listed in Table I above. The lowest loss attained was for the core blank having 2.9 wt% Ta_2O_5. At 1550 nm the attenuation was 1.73 dB/km. These results confirm the importance of using consolidation temperatures higher than 1450°C for Ta_2O_5-SiO_2 containing optical fibers. Based upon this information and the experiments described above in Examples 1 through 4, it is anticipated that Ta_2O_5-SiO_2 containing optical fibers will exhibit losses of less than about 0.25 dB/km at 1550 nm when the soot blanks corresponding to these fibers are consolidated in consolidation furnaces capable of sustaining temperatures greater than 1500°C.

It will be apparent to those skilled in the art that modifications and variations can be made to the present invention without departing from the spirit or scope of the invention. Thus it is intended that the present invention cover the modifications and variations of this invention provided they come within the scope of the appended claims and their equivalents.

WE CLAIM:

1. A method of manufacturing a low loss optical waveguide having a high refractive index core, said method comprising the steps of:

forming a soot blank comprising Ta_2O_5 and SiO_2;

consolidating said soot blank to form a cane under conditions suitable to prevent crystallization in said blank; and

drawing said blank into an optical fiber.

2. The method as claimed in claim 1 wherein the step of consolidating said soot blank comprises the steps of:

exposing said soot blank to an atmosphere comprising helium; and heating said soot blank to a temperature greater than 1550° C.

3. The method as claimed in claim 1 wherein the step of consolidating said soot blank comprises the steps of:

exposing said soot blank to a vacuum atmosphere, and

heating said soot blank to a temperature greater than 1450° C.

4. The method as claimed in claim 3 wherein the vacuum atmosphere comprises a pressure of less than about 10^{-4} torr.

5. The method as claimed in claim 2 wherein the atmosphere comprises helium and oxygen.

6. The method as claimed in claim 1 wherein the step of forming a soot blank comprises the step of doping said soot blank with between about 2.5 wt% Ta_2O_5 to about 3.5 wt% Ta_2O_5.

7. The method as claimed in claim 1 wherein said forming and consolidating steps comprise selecting parameters suitable to result in the optical fiber exhibiting a loss of less than about 1.8 dB/km at 1550 nm.

8. The method as claimed in claim 1 wherein said forming and consolidating steps comprise selecting parameters suitable to result in the optical fiber exhibiting a loss of approximately .25 dB/km at 1550 nm.

9. The method as claimed in claim 8 wherein the step of consolidating said soot blank comprises the steps of:

exposing said soot blank to an atmosphere comprising helium; and heating said soot blank to a temperature greater than 1550° C.

10. The method as claimed in claim 8 wherein the step of consolidating said soot blank comprises the steps of:

exposing said soot blank to a vacuum atmosphere; and heating said soot blank to a temperature greater than 1450° C.

11. The method as claimed in claim 1 further comprising the step of overcladding said blank to form a cladding comprising SiO_2.

12. The method as claimed in claim 1 wherein the step of forming said soot blank comprises the steps of:

flowing Cl_2 gas over Ta within a Cl_2 reactor at a temperature greater than 350° C to form $TaCl_5$;

delivering the $TaCl_5$ to an OVD burner to form soot comprising Ta_2O_5; and

depositing said soot on a rotating mandrel to form said soot blank.

13. An optical fiber made by the method of claim 1.

14. An optical fiber comprising;

a high purity glass cladding; and

a glass core bounded by said cladding, said glass core having a higher refractive index than said cladding, said glass core including between about 2-

5 wt% Ta_2O_5 after consolidation, and wherein light attenuation in said optical fiber is less than about 1.8 dB/km at 1550 nm.

15. The optical fiber as claimed in claim 14 wherein said glass core further includes SiO_2 and wherein said optical fiber is substantially free of crystals.

16. The optical fiber as claimed in claim 15 wherein light attenuation in said optical fiber comprises about 0.25 dB/km at 1550 nm.

17. A glass for use in the core of an optical waveguide comprising:

SiO_2; and

by weight on an oxide basis after consolidation, between about 2% non-crystallized Ta_2O_5 to 5% non-crystallized Ta_2O_5.

18. The glass as claimed in claim 17 wherein said core glass is consolidated in a helium atmosphere at a temperature of between about 1600° C to about 2000° C.

19. The glass as claimed in claim 18 wherein said core glass is consolidated in a helium atmosphere at a temperature of between about 1600° C to about 1800° C.

20. The glass as claimed in claim 19 wherein said core glass is consolidated in a helium atmosphere at a temperature of between about 1600° C to about 1650° C.

21. The core glass as claimed in claim 17 wherein said core glass is consolidated in a vacuum atmosphere at a temperature greater than about 1450° C.

22. The core glass as claimed in claim 17 wherein said core glass is bounded by a cladding comprising SiO_2 to form an optical fiber, and wherein light attenuation in said optical fiber is less than about 1.8 dB/km at 1550 nm.

23. The core glass as claimed in claim 22 wherein light attenuation in said optical fiber is less than 0.25 dB/km at 1550 nm.

ABSTRACT OF THE DISCLOSURE

The present invention is directed to low loss optical waveguides doped with tantala and methods of manufacturing such waveguides. SiO_2 soot is doped with Ta_2O_5 to form a soot blank which is consolidated under conditions suitable to prevent the crystallization within the Ta_2O_5-SiO_2 containing waveguides. The resulting cane is then either drawn into an optical fiber or overclad and subsequently drawn into an optical fiber. High temperature consolidation in either a gaseous atmosphere or vacuum atmosphere is used to sinter and vitrify the soot blank prior to drawing to produce a low loss optical waveguide fiber.

FIG. 1

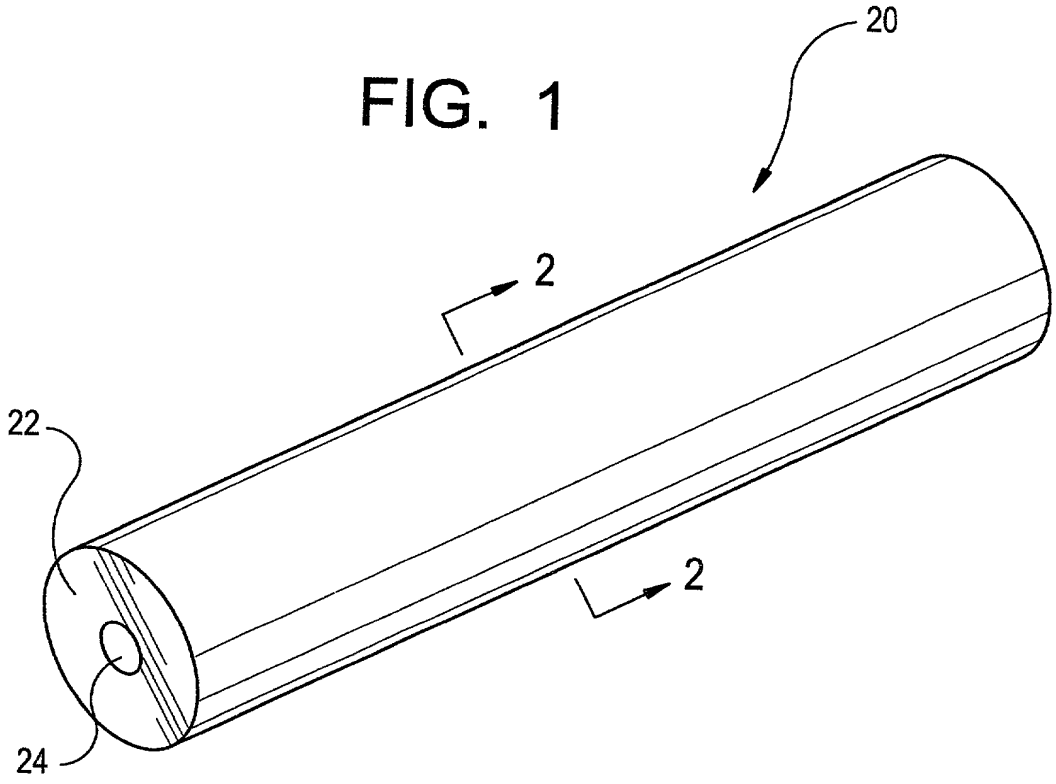


FIG. 2

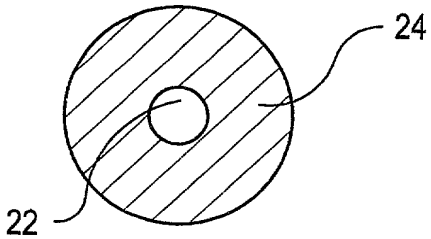


FIG. 3

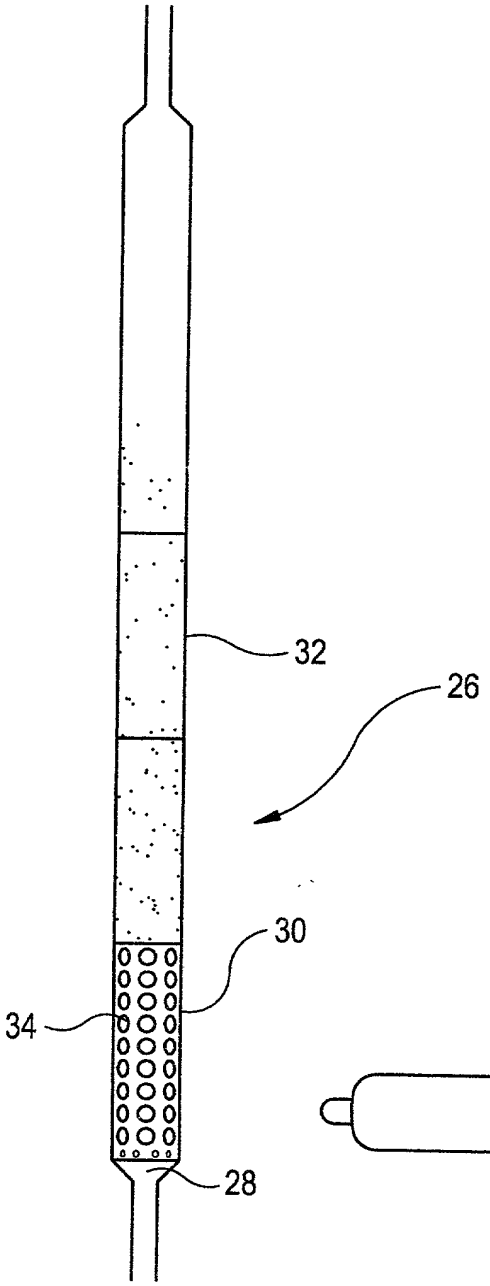


FIG. 4

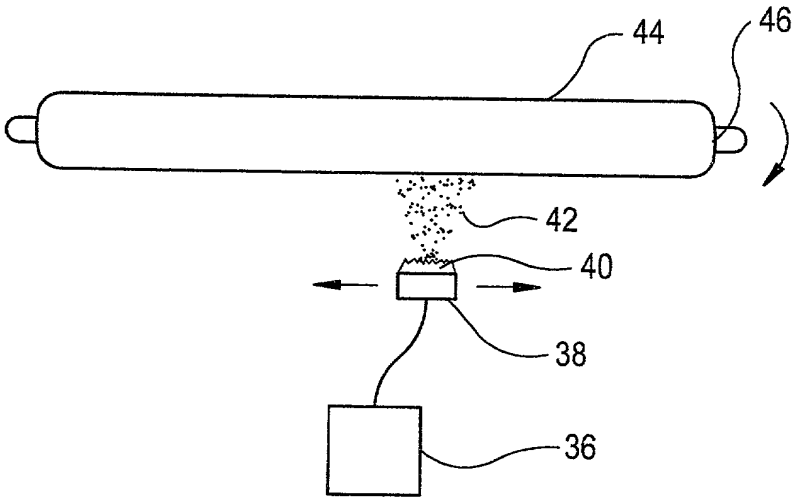


FIG. 5

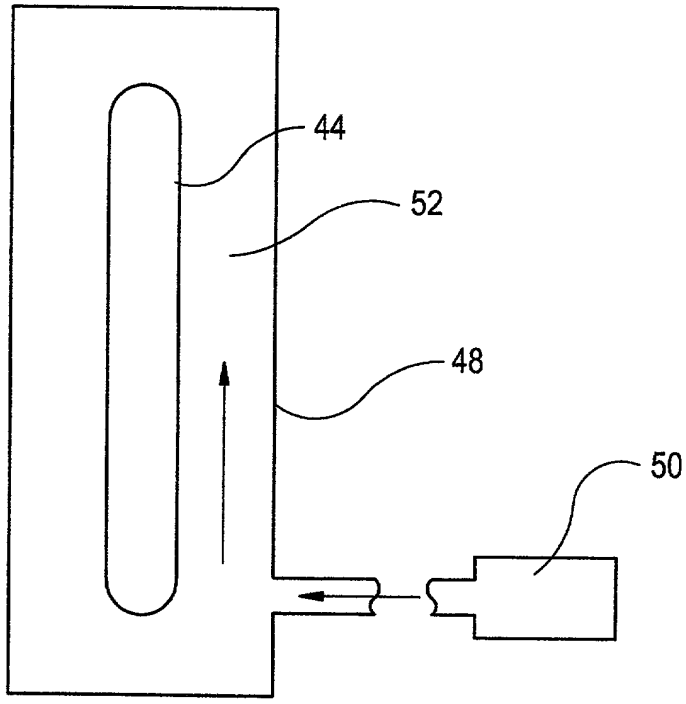


FIG. 6

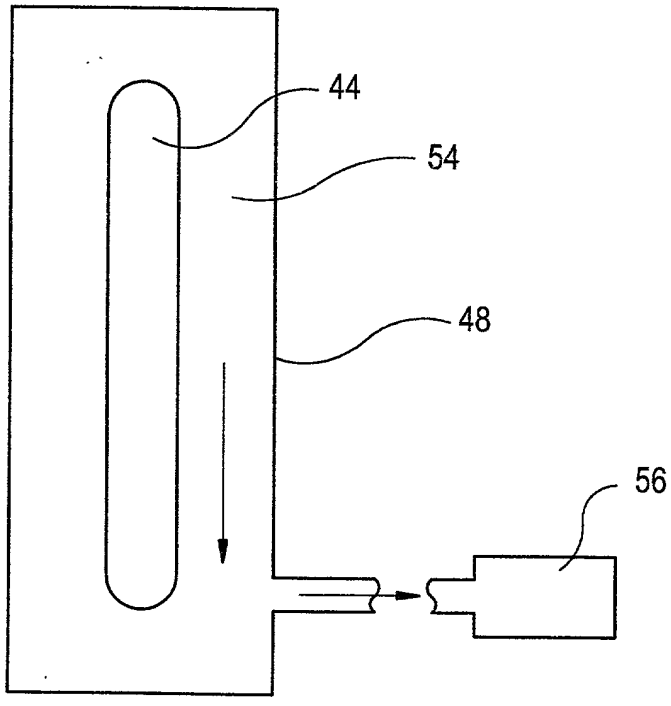


FIG. 7

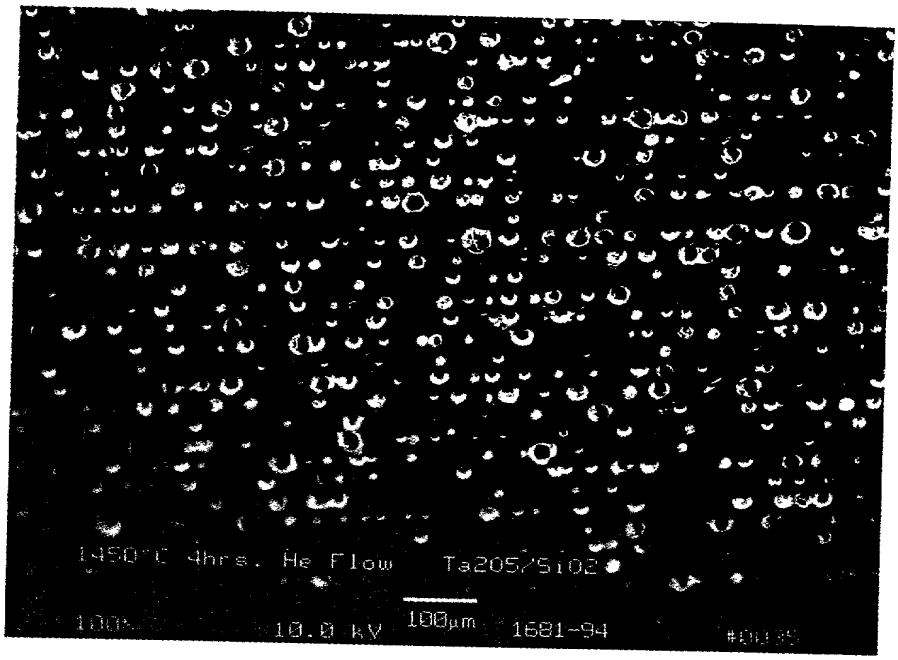

1450°C 4hrs. He Flow Ta2O5/SiO2
100µm
10.0 kV
1681-94

FIG. 8

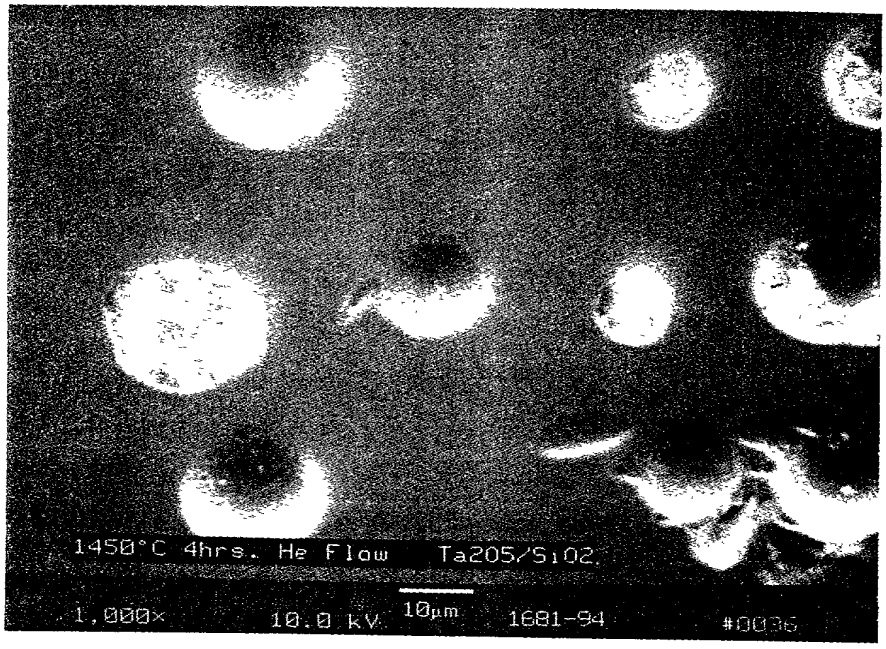

1450°C 4hrs. He Flow Ta2O5/SiO2
10µm
1,000×
10.0 kV
1681-94

FIG. 9

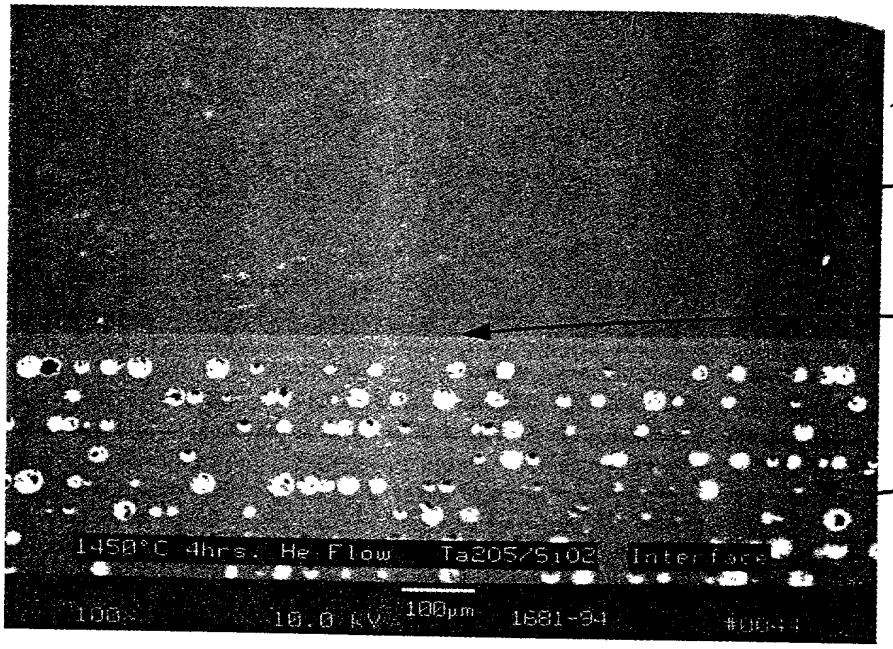

60
62
66
64
1450°C 4hrs. He Flow Ta2O5/SiO2 Interface
100x 10.0 kV 100µm 1681-94 #0041

FIG. 10

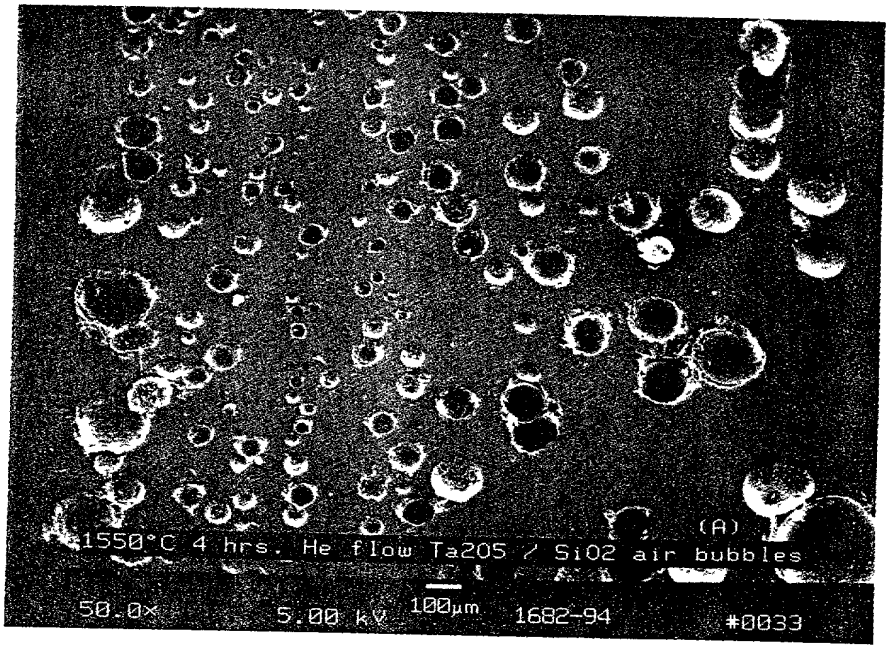

(A)
1550°C 4 hrs. He flow Ta2O5 / SiO2 air bubbles
50.0x 5.00 kV 100µm 1682-94 #0033

FIG. 11

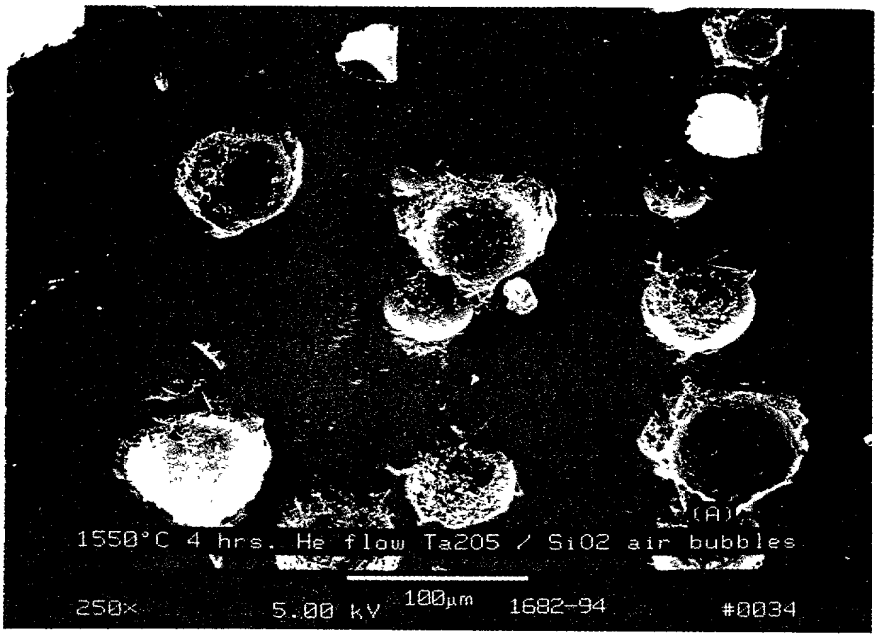

(A)
1550°C 4 hrs. He flow Ta2O5 / SiO2 air bubbles
250×
5.00 kV
100µm
1682-94
#0034

FIG. 12

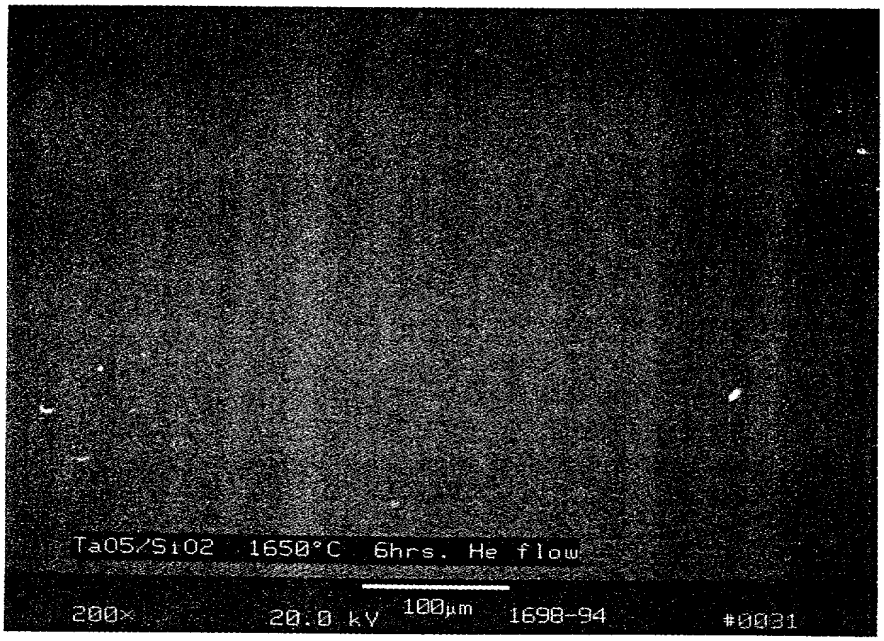

Ta2O5/SiO2 1650°C 6hrs. He flow
200×
20.0 kV
100µm
1698-94
#0031

FIG. 13

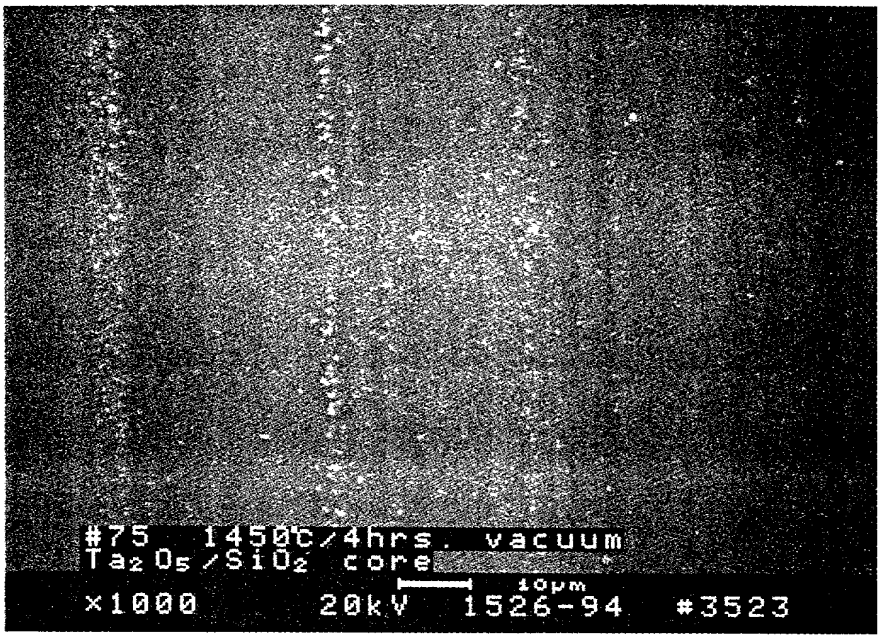

#75 1450C/4hrs. vacuum
Ta2 Os /SiO2 core
10μm
×1000 20kV 1526-94 #3523

FIG. 14

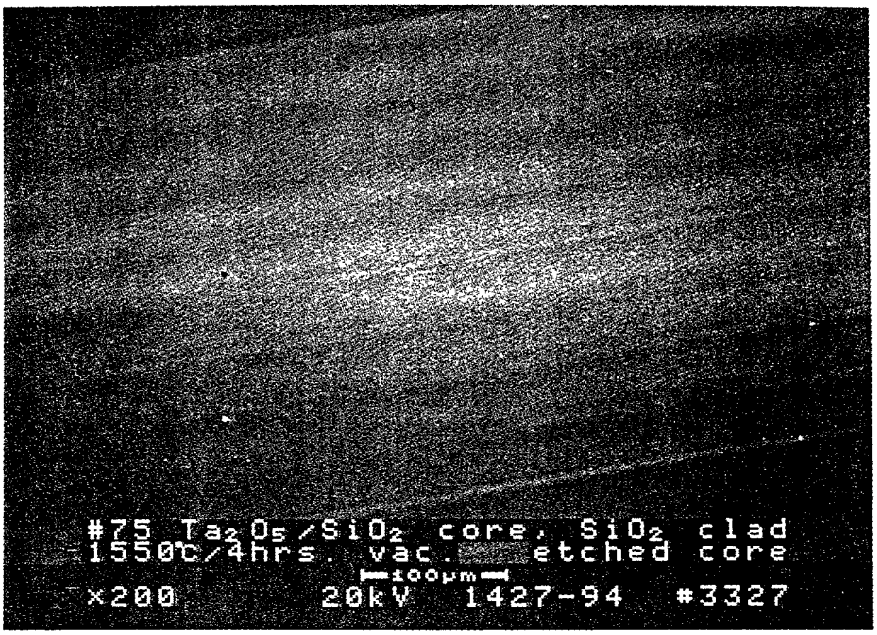
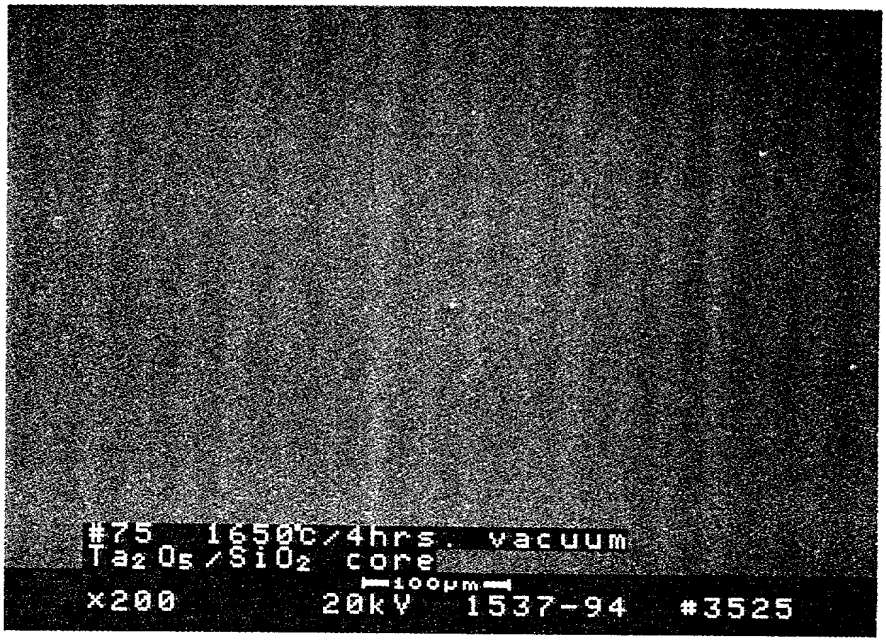

#75 Ta2 Os /SiO2 core, SiO2 clad
1550C/4hrs. vac. etched core
100μm
×200 20kV 1427-94 #3327

FIG. 15


#75 1650C/4hrs. vacuum
Ta2O5/SiO2 core
100μm
x200 20kV 1537-94 #3525

DECLARATION IN ORIGINAL APPLICATION **Attorney Docket No. DeLiso 11-3-5-8**

As a below named inventor, I declare that:

My residence, Post Office address and citizenship are as stated below next to my name.

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled **TANTALA DOPED WAVEGUIDE AND METHOD OF MANUFACTURE**, the specification of which is attached hereto.

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to the examination of this application in accordance with Title 37, Code of Federal Regulations, §1.56(a).

I hereby claim foreign priority benefits under Title 35, United States Code, §119 of any foreign application(s) for patent or inventor's certificate listed below and have also identified below any foreign application for patent or inventor's certificate, on the same subject matter, having a filing date before that of the application on which priority is claimed:

☐ **Country:** **Application No.:** **Filing Date:**

☒ **NONE**

I hereby claim the benefit under Title 35 United States Code §119(e) and §120 of any United States application(s) listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States application in the manner provided by the first paragraph of Title 35 United States Code §112, I acknowledge the duty to disclose material information as defined in Title 37 Code of Federal Regulations, §1.56(a) which occurred between the filing date of the prior application and the national or PCT international filing date of this application:

☒ **Provisional No.**: 60/114,369 **Filed**: December 30, 1998 **Status:** Pending

☐ **Application No.** ***(if a Continuation)*****:** 08/ **Filed**: **Status**: Pending

☐ **NONE**

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

DECLARATION IN ORIGINAL APPLICATION **Attorney Docket No. DeLiso 11-3-5-8**

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Christine Louise Tennent

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